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Indian Standard

SPECIFICATION FOR SODIUM METABISULPHITE, PHOTOGRAPHIC GRADE

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
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Indian Standard

SPECIFICATION FOR SODIUM METABISULPHITE, PHOTOGRAPHIC GRADE

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Indian Standard

SPECIFICATION FOR SODIUM METABISULPHITE, PHOTOGRAPHIC GRADE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 30 June 1986, after the draft finalized by the Photographic Materials Sectional Committee had been approved by the Chemical Division Council.

0.2 The specification for sodium metabisulphite, photographic grade was earlier covered under IS : 248-1978* along with technical grade of the material. The committee responsible for formulation of this standard felt that a separate standard for the photographic grade of the material should be formulated. Consequently, this standard covering only the photographic grade of the material has been formulated and the photographic grade is being deleted from IS : 248-1978*. In this standard, the requirements for matter insoluble in water and thiosulphate content for photographic grade have been modified from the requirements given for photographic grade in IS : 248-1978*.

0.3 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960†. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for sodium metabisulphite, photographic grade.

*Specification for sodium bisulphite (sodium metabisulphite) (*first revision*).
†Rules for rounding off numerical values (*revised*).

2. REQUIREMENTS

2.1 Description — The material shall be dry, white or cream coloured powder, free from extraneous matter and having a faint smell of sulphur dioxide gas.

2.2 The material shall also conform to the requirements laid down in Table 1, when tested in accordance with the methods prescribed in Appendix A.

TABLE 1 REQUIREMENTS FOR SODIUM METABISULPHITE, PHOTOGRAPHIC GRADE

(*Clauses* 2.2, A-6.3.1, A-7.3.1 *and* A-9.3.1)

Sl No.	Characteristic	Requirement	Method of Test (Ref to Cl No. in Appendix A)
(1)	(2)	(3)	(4)
i)	Purity (as SO_2 content), percent by mass, *Min*	64·0	A-2
ii)	*p*H of 5 percent solution	4·0 to 4·6	A-3
iii)	Matter insoluble in water, calcium, magnesium and ammonium hydroxide precipitates, percent by mass, *Max*	0·5	A-4
iv)	Matter insoluble in water, percent by mass, *Max*	0·02	A-5
v)	Iron (as Fe), percent by mass, *Max*	0·005	A-6
vi)	Heavy metals (as Pb), percent by mass, *Max*	0·005	A-7
vii)	Appearance of solution	To pass test	A-8
viii)	Thiosulphate content (as $Na_2S_2O_3$), percent by mass, *Max*	0·05	A-9
ix)	Reaction to ammoniacal silver nitrate	To pass test	A-10

3. PACKING AND MARKING

3.1 Packing — The material shall be suitably packed taking care that it does not come in direct contact with iron container either during storage or in transit.

3.2 Marking — The containers shall be marked with the following:

a) Name of the material;

b) Name of the manufacturer and recognized trade-mark, if any;

c) Net mass; and

d) Month and year of manufacture.

3.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SCALE OF SAMPLING AND CRITERIA FOR CONFORMITY

4.1 The scale of sampling and criteria for conformity of the material to the standard shall be as prescribed in Appendix B.

APPENDIX A

(*Clause* 2.2; and *Table* 1)

METHODS OF TEST FOR SODIUM METABISULPHITE, PHOTOGRAPHIC GRADE

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF PURITY OF SODIUM METABISULPHITE

A-2.1 Reagents

A-2.1.1 *Starch Solution* — Take 3 g of starch and make a paste with cold water. Pour the paste into 1 litre of boiling water and add 10 ml of one percent salicylic acid.

A-2.1.2 *Potassium Iodide* — crystals.

A-2.1.3 *Iodine Solution* — approximately 0·1 N. Dissolve in a 1 000-ml volumetric flask about 12·5 g of resublimed iodine in a concentrated solution of 35 g of potassium iodide. Make up the solution to 1 000 ml.

*Specification for water for general laboratory use (*second revision*).

A-2.1.4 *Standard Sodium Thiosulphate Solution* — 0·1 N. Dissolve about 25 g of crystallized sodium thiosulphate in 1 000 ml of recently boiled water in a volumetric flask. Standardize the solution against standard potassium dichromate or freshly standardized iodine solution, using towards the end of the reaction, starch solution as indicator. The solution shall be prepared fresh.

A-2.2 Procedure — Weigh accurately about 0·2 g of the material and add it to exactly 50 ml of standard iodine solution. Allow to stand for 5 minutes, add 1 ml of hydrochloric acid and titrate the excess of iodine with standard sodium thiosulphate solution, using starch solution as indicator. The end point of the reaction is indicated by the disappearance of blue colour. Carry out a blank titration with same amounts of reagents but without sample.

A-2.3 Calculation — Calculate the percentage of sulphur dioxide (SO_2) on the basis that 1 ml of 0·1 N iodine solution is equivalent to 0·003 203 g of sulphur dioxide (SO_2).

$$\text{Purity (as } SO_2 \text{ content), percent by mass} = 3{\cdot}203 \times \frac{(V_1 - V_2)\mathcal{N}}{M}$$

where

V_1 = volume in ml of standard thiosulphate solution required for blank,

V_2 = volume in ml of standard thiosulphate solution required for test,

$\mathcal{N}$ = normality of standard thiosulphate solution, and

M = mass in g of the material taken for the test.

A-3. DETERMINATION OF *p*H

A-3.1 Apparatus

A-3.1.1 *p*H *Meter* — equipped with a standard calomel electrode and a glass electrode.

A-3.2 Procedure — Dissolve 5 g of the material in 100 ml of carbon dioxide-free water. Determine the *p*H by means of the *p*H meter.

A-4. DETERMINATION OF MATTER INSOLUBLE IN WATER, CALCIUM, MAGNESIUM AND AMMONIUM HYDROXIDE PRECIPITATES

A-4.1 Apparatus

A-4.1.1 *Platinum Dish*

A-4.2 Reagents

A-4.2.1 *Ammonium Oxalate Solution* — 4 percent (*m/v*).

A-4.2.2 *Ammonium Phosphate Solution* — 10 percent (*m/v*).

A-4.2.3 *Ammonium Hydroxide Solution* — 10 percent (*m/v*).

A-4.2.4 *Ammonium Hydroxide Solution* — 2·5 percent (*m/v*).

A-4.3 Procedure — Dissolve 10·0 ± 0·1 g of sample in 75 ml of water. Add 10 ml of ammonium oxalate solution, 4 ml of ammonium phosphate solution and 20 ml of ammonium hydroxide solution. Allow to stand overnight. If any precipitate is formed, filter through filter paper (Whatman No. 42 or equivalent) and wash with 2·5 percent ammonium hydroxide solution. Ignite the filter paper with precipitate in a platinum dish at 600°C till all carbon has gone; cool and weigh.

A-4.4 Calculation

$$\text{Matter insoluble in water, calcium, magnesium, and ammonium hydroxide precipitates, percent by mass} = \frac{100 \times m}{M}$$

where

m = mass in g of the residue, and

M = mass in g of the sample taken for the test.

A-5. DETERMINATION OF MATTER INSOLUBLE IN WATER

A-5.1 Apparatus

A-5.1.1 *Gooch Crucible or Sintered Glass Crucible* — Porosity No. 4.

A-5.2 Procedure — Weigh accurately about 50 g of the material and dissolve in 300 ml of water. Filter through the tared Gooch crucible or tared sintered glass crucible and wash well with water. Dry at 105 ± 2°C to constant mass.

A-5.3 Calculation

$$\text{Matter insoluble in water, percent by mass} = \frac{100 \times m}{M}$$

where

m = mass in g of the residue obtained, and

M = mass in g of the sample taken for the test.

A-6. TEST FOR IRON

A-6.1 Apparatus

A-6.1.1 *Nessler Cylinders* — 50-ml capacity.

A-6.2 Reagents

A-6.2.1 *Ammonium Persulphate* — solid.

A-6.2.2 *Concentrated Hydrochloric Acid* — *See* IS : 265-1976*.

A-6.2.3 *Butanolic Potassium Thiocyanate Solution* — Dissolve 10 g of potassium thiocyanate in 10 ml of water. Add sufficient *n*-butanol to make 100 ml and shake vigorously until solution is clear.

A-6.2.4 *Standard Iron Solution* — Dissolve 7·020 g of ferrous ammonium sulphate [$FeSO_4.(NH_4)_2SO_4.6H_2O$] in water containing 10 ml of dilute sulphuric acid (10 percent) and dilute to one litre in a volumetric flask. One millilitre of the solution contains 0·1 mg of iron (as Fe). Further dilute 100 ml of the solution to 1 000 ml. One millilitre of the diluted solution contains 0·01 mg of iron (as Fe).

A-6.3 Procedure — Dissolve 1·000 g of the sample in 30 ml of hot water, add 5 ml of hydrochloric acid and evaporate to dryness on a steam bath. Add 15 ml of hot water and 2 ml of hydrochloric acid and evaporate again to complete dryness. Dissolve the residue in 10 ml of water and transfer to a beaker. Add 1 ml of hydrochloric acid, about 30 mg of ammonium persulphate, heat to boiling, cool and transfer to a Nessler cylinder. Add 15 ml of butanolic potassium thiocyanate. Shake vigorously for 30 seconds and allow the two layers to separate. For control test, take 5 ml of standard iron solution and repeat the test as above.

A-6.3.1 The limits prescribed in Table 1 shall be taken as not having been exceeded if the red colour produced in the butanolic layer of the sample solution is not more intense than that produced in the control test.

A-7. TEST FOR HEAVY METALS

A-7.1 Apparatus

A-7.1.1 *Nessler Cylinders* — 50 ml capacity.

A-7.2 Reagents

A-7.2.1 *Acetic Acid* — approximately 33 percent (*m/v*).

A-7.2.2 *Dilute Ammonium Hydroxide Solution* — approximately 10 percent (*v/v*).

A-7.2.3 *Concentrated Hydrochloric Acid* — *See* IS : 265-1976*.

*Specification for hydrochloric acid (*second revision*).

A-7.2.4 *Standard Lead Solution* — Dissolve 1·60 g of lead nitrate in water and make up the solution to 2 litres in a volumetric flask. One millilitre of the solution contains 0·5 mg of lead (as Pb). Further dilute 100 ml of the solution to 1 000 ml. One millilitre of the diluted solution contains 0·05 mg of lead (as Pb).

A-7.2.5 *Sodium Sulphide Solution* — 10 percent (*m/v*), freshly prepared.

A-7.3 Procedure — Dissolve 4·000 g of the sample in 20 ml of hot water. Add 6 ml of hydrochloric acid and evaporate the contents nearly to dryness on a water bath. Add 15 ml of hot water, 3 ml of hydrochloric acid and evaporate again on water bath. Finally heat for 1 hour at 150°C. Dissolve the residue in water, filter and make up the filtrate to 50 ml in a volumetric flask. Pipette into a Nessler cylinder 12·5 ml of the sample solution, 2 ml of standard lead solution and 1 ml of acetic acid. Add 2 drops of sodium sulphide solution and make up to the mark with water. Mix well. To another Nessler cylinder, add the remaining 37·5 ml of the sample solution and 1 ml of acetic acid. Add 2 drops of sodium sulphide solution and make up to the mark with water. Mix well.

A-7.3.1 The limits prescribed in Table 1 shall be taken as not having been exceeded if the colour produced in the second cylinder is not more intense than that produced in the first.

A-8. APPEARANCE OF SOLUTION

A-8.1 Dissolve 20 g of the material in a little quantity of water and make up the volume to 100 ml. The solution shall not have more than a pale yellow colour and shall be free from extraneous impurities other than slight flocculence.

A-9. TEST FOR THIOSULPHATE

A-9.1 Apparatus

A-9.1.1 *Nessler Cylinders* — 50-ml capacity.

A-9.2 Reagents

A-9.2.1 *Potassium Bromide-Mercuric Chloride Reagent* — Dissolve 25 g each of potassium bromide and mercuric chloride in 900 ml of water at a temperature of about 50°C. Cool, dilute to 1 litre and allow to stand overnight. Filter, if necessary, to obtain a perfectly clear solution.

A-9.2.2 *Standard Sodium Thiosulphate Solution* — Dilute 5·0 ml of exactly 0·1 N sodium thiosulphate ($Na_2S_2O_3.5H_2O$) solution (*see* IS : 2316-1968*) to 1 litre.

*Methods of preparation of standard solutions for colorimetric and volumetric analysis (*first revision*).

A-9.3 Procedure — Dissolve 5·0 ± 0·1 g of the material in water and dilute to exactly 100 ml. Slowly pipette 1 ml of the solution into a Nessler cylinder containing 10 ml of potassium bromide-mercuric chloride reagent. In a second Nessler cylinder containing 10 ml of potassium bromide-mercuric chloride reagent, add 0·3 ml of standard sodium thiosulphate solution. Let both the cylinders stand for 15 minutes without agitation. Then carefully agitate to distribute the opalescence and examine immediately.

A-9.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the opalescence produced with the material is not greater than that in the control test.

A-10. REACTION TO AMMONIACAL SILVER NITRATE SOLUTION

A-10.1 Apparatus

A-10.1.1 *Nessler Cylinders* — 50-ml capacity.

A-10.2 Reagents

A-10.2.1 *Ammoniacal Silver Nitrate* — Take equal volumes of strong ammonia and 10 percent (*m/v*) silver nitrate. When required, the solution shall be prepared fresh.

A-10.3 Procedure — Weigh, to the nearest 0·1 g, about 2 g of the material and dissolve in 40 ml of water. Divide into two equal portions. To one, add 10 ml of ammoniacal silver nitrate solution and mix well. To the other, the control solution, add 5 ml of ammonia solution and 5 ml of water and mix well. Allow each to stand for 2 min. Compare the colours and turbidities of the two solutions.

A-10.3.1 The material shall be taken to have passed the test if the colour or the turbidity produced with the material is not greater than that produced in the control test.

APPENDIX B

(*Clause* 4.1)

SAMPLING OF SODIUM METABISULPHITE, PHOTOGRAPHIC GRADE

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling test samples, the precautions given in **B-1.1** to **B-1.7** shall be observed.

B-1.1 Samples shall not be taken at a place exposed to the weather.

B-1.2 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.3 To draw a representative sample, the contents of each container selected for sampling shall be mixed thoroughly by suitable means.

B-1.4 The samples shall be placed in suitable, clean, dry and air-tight opaque glass or plastic containers.

B-1.5 The sample containers shall be of such a size that they are almost completely filled by the sample.

B-1.6 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling and the month and year of manufacture.

B-1.7 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

B-2. SCALE OF SAMPLING

B-2.1 Lot — All the containers in a single consignment of the material and drawn from a single batch of manufacture shall constitute a lot.

B-2.1.1 Samples shall be tested from each lot for ascertaining the conformity of the material to the requirements of the specification.

B-2.2 The number (n) of containers to be chosen from a lot depends on the size of the lot (N) and shall be in accordance with col 1 and 2 of Table 2.

TABLE 2 SCALE OF SAMPLING OF CONTAINERS

Lot Size	Number of Containers to be Selected in the Sample
N	n
(1)	(2)
Up to 50	3
51 to 100	4
101 to 300	5
301 and above	7

B-2.3 The containers to be selected for sampling shall be drawn at random from the lot. For random sampling procedures, guidance may be obtained from IS : 4905-1968*.

*Methods for random sampling.

B-3. PREPARATION OF TEST SAMPLES

B-3.1 Draw with an appropriate sampling instrument 50 g of sodium metabisulphite from different parts of each container selected. This portion shall be transferred to suitable sample container. From each of the sample containers, approximately equal quantities of the material shall be taken and mixed together to give a composite sample weighing about 100 g. The remaining material in each of the sample container is termed as individual sample.

B-4. CRITERIA FOR CONFORMITY

B-4.1 Test for purity of the material shall be conducted on individual samples. The lot shall be considered as conforming to the specification if all the individual samples pass the test.

B-4.2 Tests for all other characteristics shall be carried out on composite sample. The lot shall be declared as conforming to the specification if the test results on the composite sample satisfy the corresponding requirements.